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Indian Standard

TESTING OF PHOSPHATE COATINGS — METHODS

ICS 77.060; 25.220.60

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 2

Corrosion Protection Sectional Committee, MTD 24

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee had been approved by the Metallurgical Engineering Divison Council.

Considerable number of different phosphating processes are in use. The purposes for which they are employed are also very large. Bureau of Indian Standards has already prepared two Indian Standards namely IS 6005 : 1970 (*under revision*) and IS 3618 : 1966 (*under revision*). Earlier the methods of tests of phosphate coatings were covered in IS 3618 : 1966. It has been decided to formulate a separate Indian Standard for methods of testing of phosphate coatings.

In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries, in addition to the practices being followed in this country. This has been met by deriving assistance from the following publications:

i) DIN 50947 : 1973 Phosphating of metal process principles, symbols and test method

ii) MIL- P-50002A : 1968 Phosphate coating compound, for phosphating ferrous metals

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standards

TESTING OF PHOSPHATE COATINGS — METHODS

1 SCOPE

This standard covers the methods for determination of mass, corrosive residues, resistance, to corrosion and composition of phosphate coatings.

- a) Test for mass of coating,
- b) Test for corrosive residue,
- c) Test for resistance to corrosion, and
- d) Qualitative testing of the coating composition.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
3531 : 1997	Glossary of terms relating to corrosion of metals

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 3531 shall apply.

4 DETERMINATION OF MASS OF COATING

4.1 The mass of the phosphate coating should be determined by the following method, on samples which have not received any finishing treatment.

4.2 The phosphated panel shall be of suitable size (approximately 5 cm × 10 cm). It shall be numbered before weighing for identification and shall be thoroughly degreased in trichloroethylene vapour or clean mineral spirits. The panel (after drying, if necessary and cooling in a desiccator to room temperature) shall be weighed to the nearest 0.1 mg and this mass shall be recorded as X g.

4.3 The panel shall then be immersed in freshly prepared solution of concentrated hydrochloric acid with antimony trioxide as an inhibitor in the proportion of 20 g per 100 ml of acid. The temperature of the stripping bath shall be maintained between 25°C and 35°C. After the coating is dissolved (a period of 5 min is normally sufficient),the component shall be removed from the acid, washed for 30 s in running water. Any non-adherent matter shall be removed with a soft wet brush.The panel shall then be dried with a clean absorbent material (Lambswool) placed in a desiccator for 10 min and re-weighed to the nearest 0.1 mg. This mass shall be recorded as Y g.

4.4 Repeat the steps outlined under **4.3** until two successive readings do not differ by more than 0.5 mg. The final mass Y g shall be used to calculate mass of coating in accordance with the formula given under **4.6**.

4.5 The total panel area shall include both sides of the panel, if both are coated.

4.6 Calculate the mass of coating as follows:

$$\text{Mass of coating, in g/m}^2\ A = \frac{X - Y}{A} \times 10\,000$$

where

X = initial mass, in g;
Y = finial mass in g; and
A = total coated panel area in cm^2.

4.7 A control panel having the same metal composition and of the same dimensions shall be used and the loss in mass of the panel should be applied as correction factor in the coating mass.

5 DETERMINATION OF CORROSIVE RESIDUES

5.1 This test is conducted to check the efficiency of the rinsing procedure. Samples of the sealed components or test panels shall be subjected to the following test, at the end of which they shall show no rust or other defect, for example, blistering or flaking of the organic coating. Where the organic coating is a multiple coat paint system, the test shall be made after the final coat has been applied.

5.1.1 Before testing, all samples shall be conditioned for 24 h at room temperature (27 ± 5°C), protected from contamination.

5.2 This test indicates the presence of harmful water soluble matter under the paint film, and is particularly valuable for showing contamination by finger marks or through splashing.

5.3 *Test Procedure*

The sample shall be suspended for 7 days at a temperature between 25 to 35°C in a closed vessel in which air is kept humid by a layer of water at the bottom of the vessel. At the end of the test the samples shall be removed and inspected immediately for signs of rust or blistering of the paint film.

6 DETERMINATION OF RESISTANCE TO CORROSION

6.1 The resistance of the sealed components to corrosion shall be tested in the laboratory by means of the ARE salt droplet test.

6.2 Test Procedure for ARE Salt Droplet Test

The specimens shall be suspended vertically, by means of hooks of glass or other non-corrodible insulator passing through the holes in two corners, over large dishes partly filled with water. The distance between the surface of the water and the lower edges of the specimens shall be 50 to 75 mm and the specimens shall be one behind the other and all face the same way. They shall be at a convenient distance apart (nor more than 25 mm) to prevent touching when being moved and a dummy specimen shall be placed at each end of each row.

6.2.1 Once daily for 5 days a week, all the specimens, including the dummies, shall be removed temporarily from their position on the rack and sprayed individually on both sides with the spray solution (*see* **6.3**), using a hand-operated atomiser (*see* **6.4**). The aim shall be to cover the surface with discrete droplets, by giving a number of puffs, with the nozzle at a suitable distance (150 mm to 300 mm) from the specimen, the spray being directed in turn at different parts of the suface. The spraying shall not be heavy that the droplets coalesce; in order to avoid this it may prove necessary on occasions, where the surface is still wet from the previous spraying to refrain from applying the full mass of spray specified below. It is essential to keep the specimen covered with droplets.

6.2.2 Immediately after spraying, the specimens shall be returned to their positions on the rack and the whole collection shall be covered with a box like covers of sheet metal or other suitable material to prevent the drying of the droplets (*see* Note under **6.2.5**).

6.2.3 Check the intensity of sparying by weighing the solution deposited on both sides of a blank (150 mm × 100 mm) bare steel specimen, freshly degreased. The mass should lie between 0.5 g and 1.0 g.

6.2.4 Any cover shall be suitably protected on the inside to prevent contamination of the specimen.

6.2.5 The temperature throughout the test shall preferably be between 25 and 35°C and the test assembly shall be protected against external sources of heat, tending to produce local variations in temperature.

NOTE — The spray may be absorbed by non-glossy paint films, if so, it may be impossible to check if the desired distribution of droplets has been obtained. This difficulty may be overcome by spraying dummy specimens of a material on which the droplets may be seen in such a way as to produce the standard distribution of droplets and then spraying the test specimens in exactly the same way. Steel or glass coated with a hard glossy finish is suitable for the purpose.

6.3 Spray Solution

6.3.1 The spray solution shall be a synthetic sea-water, made by dissolving the following salts in distilled water:

Salt	*Concentration* g/l
Sodium chloride, NaCl	23
Sodium sulphate, $Na_2SO_4.10H_2O$	8.9
Magnesium chloride, $MgCl_2 . 6H_2O$	9.8
Calcium chloride (anhydrous), $CaCl_2$	1.4

6.3.1.1 The salts used shall be of analytical grade.

6.4 Atomizer

Any atomizing nozzle capable of producing fine droplets (not a mist) may be used for the test. The whole apparatus including the container shall be made of non-metallic material, for example, glass or ebonite.

6.5 Loss in Mass of Control Specimens

6.5.1 At the end of the agreed test period, the loss in mass of the control specimens shall be detemined after derusting as described in **6.5.1.1** and **6.5.2**.

6.5.1.1 The specimens shall be derusted by means of Clarke's solution which consists of 20 g of antimonious oxide and 50 g of stannous chloride dissolved in one litre of hydrochloric acid (r.d. 1.16). The solution shall be used cold and the specimen kept moving in it until derusting is complete. The specimens shall then be washed in running water, dried and weighed. The figure to be recorded is the difference between the mass of the freshly degreased specimen and the corresponding mass after derusting.

It is essential that the specimens be kept moving in this solution so as to ensure rapid reduction by the reagent of ferric chloride produced by solution of the rust, otherwise attack on the steel itself may occur.

6.5.2 The average loss in mass shall not be less than the values given in Table 1.

Table 1 Average Loss in Mass

Test Duration, Days	Average Loss in g in Mass per Specimen of Size 150 mm × 100 mm
(1)	(2)
1	0.2
2	0.6
3	1.5
5	3.8
7	4.7

6.6 Inspection

At the end of the agreed test period the specimens shall be rinsed in cold water, visually inspected, and allowed to dry at room temperature. Immediately after the specimens are dry, a final inspection shall be carried.

The same procedure shall be followed if failure is suspected at an earlier stage, and if inspection confirms failure, the specimen or specimens concerned may be removed from test.

7 QUALITATIVE TESTING OF THE COATING COMPOSITION

7.1 Apart from the analytical methods indicated in this standard, all other methods of analysis which are at least equal to them in sensitivity are permitted. For the identification of the coatings as phosphate coatings it is sufficient to verify phosphate in the coating (*see* **7.4**).

To identify phosphate coatings as manganese phosphate, zinc phosphate, zinc calcium phosphate or zinc magnesium phosphate coatings, the coating shall be tested for manganese according to **7.5** in the case of ferrous materials, and for zinc according to **7.6** in the case of zinc-free materials. If the test according to **7.5** reveals manganese in the coating, the coatings concerned are of manganese phosphate.

If the test according to **7.5** reveals zinc in the coating, it is necessary to make a further test according to **7.6** for calcium in the coating in order to permit differentiation between zinc phosphate coatings and zinc calcium phosphate coatings.

7.2 Quality of Reagents

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the test.

7.3 Reagents

All the reagents shall be of analytical grade.

7.3.1 *Sodium Hydroxide Solution*, approximately 5 percent (*m/v*).

7.3.2 *Dilute Nitric Acid*, 40 percent (*v/v*), 1:4 (*v/v*).

7.3.3 *Ammonium Molybdate Solution*

Take 88.3 g of ammonium molybdate, 30-ml aqueous ammonia solution with (25 percent) NH_3 and 240 g of ammonium nitrate dissolved in distilled water in one-litre volumetric flask and made the volume to the mark.

7.3.4 *Hydrochloric Acid*,25 percent (*v/v*), 1:3 (*v/v*).

7.3.5 *Potassium Hexaferrocyanate* (II) $K_4Fe(CN)_6$, 5 percent (*m/v*).

7.3.6 *Hydrogen Peroxide*, 3 percent (*v/v*).

7.3.7 *Sodium Bismuthate*, solid.

7.3.8 *Ammonium Nitrate*, solid.

7.3.9 *Aqueous Ammonia Solution*, 1:2 (*v/v*).

7.3.10 *Ammonium Oxalate*, $(NH_4)_2C_2O_4\ H_2O$.

7.3.11 *Ammonium Chloride* (NH_4Cl), solid.

7.3.12 *Sulphuric Acid*, 1:2 (*v/v*).

7.3.13 *Potassium Permanganate Solution*, approximately 5 percent (*m/v*).

7.3.14 *Quinalizarin*

Prepare by dissolving 0.01 - 0.02 g of quinalizarin in 100 ml of alcohol. 2N sodium hydroxide is prepared by dissolving 80 g of sodium hydroxide in one litre of distilled water.

7.4 Test for the Presence of Phosphate in the Coating

A specimen with a test surface measuring approximately 100 cm^2 is treated with 100 ml of 5-percent sodium hydroxide solution at 80 to 90°C until the coating has either been stripped off or has at least undergone obvious attack. If necessary the coating should be stripped off with a rubber squeeze. The resulting solution is then filtered. 25 ml of the filterate is acidified with ammonium molybdate reagent and approximately 5 g of ammonium nitrate is then added. The specimen shall be left to stand for at least 15 min. A yellow preciptate indicates the presence of phosphate.

7.5 Test for the Presence of Manganese in the Coating

To test for the presence of manganese, the filter residue according to **7.4** is stripped from the filter with hot, approximately 17-percent nitric acid to which a few drops of 3 percent hydrogen peroxide has been added. To decompose the surplus hydrogen peroxide the solution is kept boiling for few minutes and then allowed to cool to room temperature. The solution is then divided into two approximately equal parts. Following the addition of 0.5 g of sodium bismuthate to one part of the solution, any manganese present is oxidized to permaganate. A reddish-violet colouration indicates the presence of manganese.

The other half of the solution is used for testing for presence of calcium in the coating according to **7.7** when zinc has been found in the coating by applying the test according to **7.6**.

7.6 Test for the Presence of Zinc in the Coating (Only Applicable to Phosphate Coating on Zinc-Free Materials)

To test for the presence of zinc, 25 ml of the filterate obtained according to **7.4** is lightly acidified with 25 percent hydrochloric acid. 5 ml of 5 percent potassium hexaferrocyanate (II) solution is then added. A white precipitate, which may take on a slight green colourration if there is any iron, indicates the presence of zinc.

7.7 Test for the Presence of Calcium in the Coating

For this purpose a further quantity of about 50 ml of the alkaline filtrate obtained according to 7.4 is combined with that portion of the nitrate solution from the test according to 7.4 which has been put aside for testing for calcium. The drop by drop addition of nitric acid (17 percent) establishes a clear solution, the *p*H value of which is subsequently adjusted to 2 to 3 (check by special indicator paper) by the dropwise addition of ammonia solution (approximate 10 percent). When the solution has been heated to about 80°C, 1 g of solid ammonium oxalate is then dissolved in it so that the *p*H value of the solution changes to 5 to 6 (check by special indicator paper). If the *p*H value is found to be lower, add solid ammonium chloride to establish a *p*H value of 5 to 6.

In the presence of calcium the solution, which is at about 80°C, precipitates finely crystalline white calcium oxalate which is filtered off after about 10 min and washed with hot distilled water to which some ammonium oxalate (2 g/l) has been added. For purification it is re-precipitated. For this purpose it is re-dissolved on the filter paper with 10 percent (Approximately) hot hydrochloric acid; the resulting solution, heated to about 80°C, is adjusted to *p*H of 5 to 6 by addition of approximately 10 percent ammonia solution, so that calcium is again precipitated as finely crystalline calcium oxalate. This is filtered off after 10 min and washed thoroughly with hot distilled water. The last washings shall not show any reaction with 15 percent potassium permanganate solution (*see* below for decolourization of the permanganese dripped in) after acidifying it with approximately 45 percent sulphuric acid.

The white precipitate is identified as calcium oxalate by spraying from the filter with hot distilled water, mixing with about 50 ml of distilled water, dissolving by adding 5 ml of approximately 45 percent sulphuric acid and adding 5 percent potassium permanganate solution drop by drop at about 80°C. Decolourization of the dripped-in permanganate solution identifies the precipitate as calcium oxalate and at the same time indicates the presence of calcium in the coating.

7.8 Test for the Presence of Magnesium in the Coating

Magnesium behaves similar to calcium, but magnesium oxalate is more soluble. The magnesium oxalate is precipitated in the same manner as is described in 7.7 for calcium, but care shall be taken that the precipitate is washed with as little ammonium oxalate solution as possible. A specific test for magnesium is then carried out as follows:

The oxalate crystals are dissolved in hydrochloric acid and the solution placed in a test tube or on a white test plate with cavities. Add approximately, the double volume of quinalizarin reagent and the solution is coloured yellowish-red. Add sodium hydroxide solution (2 N) untill the colour changes to violet and a further excess to increase the volume by 25 to 50 percent.

A cornflower-blue percipitate or colouration appears if magnesium is present which is readily distinguished from the blue-violet colour of the reagent.

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